

A catalyst and refractory carrier **106** is then applied by spraying a solution of the paste through an aligned shadow mask onto the active area constituted by the heater element **102**. An additional improvement is to apply the same catalyst and refractory carrier using the same method from the base of a silicon wafer **54** so as to fully coat the heater element. The mask for this step is formed by the lower surface of the wafer structure instated in FIG. 7K.

The embodiment illustrated in FIGS. 5A and 5B utilizing separate heater and temperature sensors may be manufactured using either of the methods illustrated in FIGS. 6A to 6K or 7A to 7L. Packaging of the sensors is performed using a standard procedure of the type applied to discrete electronic components such as transistors. The individual die is removed from the plastic sheet upon which the wafer was attached during dicing, and is mounted onto a standard transistor header using an epoxy cement. After the epoxy has hardened thin aluminum wire is used to connect electrically the heater element with the wire posts on the heater. A lid having a upper aperture is then welded onto the header so as to protect the sensor.

Referring now to FIGS. 8A to 8E, cross-Seton views of various stages in the manufacture of a preferred embodiment are shown. In FIG. 8A, a polished silicon wafer **110** having a (1-0-0) crystal orientation is formed having a Silicon dioxide layer **112** as a base on an operatively lower face of the silicon wafer **110** and a dielectric thin film of alumina **114** having a thickness of 0.2 to 03  $\mu\text{m}$  is deposited on the operatively upper face of the silicon wafer **110** using conventional thin film processes.

As is clear from FIG. 8B and FIG. 9, a platinum layer having a thickness of 1.8  $\mu\text{m}$  to 2.2  $\mu\text{m}$  and comprising a central meander structure **116** flanked by outer electrically conductive terminal layers **118** is deposited on top of the alumina layer **114**. The alumina layer is selected to match the thermal expansion of the platinum, thereby minimizing stress build-up between adjoining layers. The platinum layer is deposited and patterned using standard techniques developed for the micro-electronics industry. The dielectric alumina layer and the inert layer of silicon dioxide are patterned using similar techniques. The alignment of the top and bottom patterns is accomplished by infra-red light to render the silicon transparent. Other alignment techniques may also be used. After the dielectric and inert layers **112** and **114** have been etched away and patterned, as is illustrated at **120** and **122** in FIG. 8C, a cavity **124** is etched away using silicon etchants having anisotropic behavior, thereby creating a suspended meander structure **126**.

A dielectric layer **128** of alumina of 1  $\mu\text{m}$  thickness is deposited on the meander structure so as to strengthen it and to act as a barrier layer against diffusion from a catalytic layer or bead **130** which is subsequently deposited on top of the dielectric layer **138**. The catalyst is contained within a thick film paste which is deposited onto the meander structure by means of screen-printing or other thick film techniques, resulting in a final suspended sensing structure **132**. In FIG. 9, the double-O configuration defined by the platinum layer can clearly be observed, as can the meander structure, with the meander structure **132** being supported entirely on four platinum bridging leads **134A**, **134B**, **134C** and **134D** also having a thickness of 1.8  $\mu\text{m}$  to 2.2  $\mu\text{m}$ .

The gas sensitive layer is composed of a calcined activated alumina onto which a noble metal catalyst is deposited. Palladium (II) chloride or palladium (II) nitrate are commonly used as catalyst precursors. The nitrate salt does have the advantage that no chlorides, which could have an inhibiting effect, remain on the alumina.

In a preferred version of the catalyst manufacturing process, the palladium (II) chloride precursor is milled with sub-micron sized (down to diameters of a few nm) alumina to reduce the size of the palladium chloride particles, using methanol as a solvent. The ratio of palladium chloride to alumina is about 25% by weight. The high palladium content results in the metal actually binding the alumina particles together. After milling, most of the methanol is removed by evaporation, and the addition of a terpeneol/ethyl cellulose organic vehicle yields a screen-printing paste suitable for screening or stenciling. In the other methods of catalyst preparation (using silica sol, glass frit and alumina precursor or acetic acid as binders) the base material is prepared by a standard impregnation process for the deposition of the palladium precursor material on the support. The alumina powder is typically soaked for several hours in a solution of palladium (II) chloride in aqueous hydrochloric acid. The hydrochloric acid and water is then slowly evaporated.

The base material of which the compensating or reference element is composed can be the same alumina with most of its activity removed by poisoning the active sites with potassium. This is achieved by soaking alumina in a potassium hydroxide/water solution containing 5% to 10% by weight of potassium hydroxide for several hours. The water is slowly evaporated and replaced by methanol as the vehicle for screen printing. A solution of ethyl cellulose in terpeneol is also added to improve the screen printing properties of the paste. The reference elements are calcined under the same conditions as the active elements, i.e. calcination in air at 750° C. for approximately 30 minutes. This treatment has been found to remove most of the activity to hydrogen which is essential for correct operation in an atmosphere containing hydrogen gas in amounts below the lower explosive limit.

In an alternative method of poisoning, an alumina element, either with or without potassium hydroxide, is covered with a thin layer of sodium silicate glass. The sodium silicate (or water glass) is a 50 wt %  $\text{SiO}_2$  solution in water. This is diluted to a 25 wt %  $\text{SiO}_2$  solution which is then applied to the degassed reference elements. The sodium silicate may also be incorporated into the bulk of the reference elements. After a drying time, the reference elements can be cured either electrically or thermally by heating in a furnace.

In the case of the preferred version of the active elements (palladium chloride milled with alumina) an inorganic binding agent is not needed. This function is filled by palladium particles finely dispersed throughout the bulk of the element.

In order to improve the adhesion of the alumina paste to the meander, it is feasible to screen a glass frit paste (a lead-free glass frit of particle size <10 microns mixed with terpeneol or ethyl cellulose) onto the platinum meanders, subsequently drying, and then screening the alumina paste onto the glass frit and calcining in the usual way, at 750° C. for 30 minutes.

After calcination, the catalytic activity of the sensors is increased and stabilized by repeatedly heating at a higher temperature than the operating temperature in a methane/air mixture for two minutes at a time, until the activity has stabilised, as is indicated by sensitivity measurements. This process is known as conditioning.

After conditioning, the preferred version sensors yielded a sensitivity of up to 100 mV when operated in 2.5% methane in air at a voltage of 2V in a Wheatstone bridge test circuit.

There are three alternative methods in which an inorganic binding agent and the active and reference materials may be